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25 (54) [TITLE OF THE INVENTION] Method for handling  
acrylonitrile solution of maleimide

(57) [ABSTRACT]

[Object] To provide a method for safe and stable handling  
of an amide in the form of an acrylonitrile solution in the  
30 presence of a polymerization inhibitor.

[Construction] In the handling of a maleimide in the  
form of an acrylonitrile solution in the presence of a

polymerization initiator, the molecular oxygen content of the gas phase part of this solution is maintained in the range of 0.01 - 10 vol. % (on the basis of the volume of the mixed gas excluding maleimide and acrylonitrile).

5        [Effect] The method is capable of preventing the acrylonitrile solution of the maleimide from becoming turbid or forming a polymer even when the solution suffers entry therein of a minute quantity of extraneous substance such as peroxide or aromatic vinyl compound and consequently  
10        allowing stable handling of the acrylonitrile solution.

[SCOPE OF CLAIM FOR PATENT]

      [Claim 1] A method for handling a maleimide in the form of an acrylonitrile solution in the presence of a polymerization inhibitor, characterized by maintaining the  
15        molecular oxygen content of the base phase part of said acrylonitrile solution in the range of 0.01 - 10 vol. % based on the volume of the gas mixture excluding maleimide and acrylonitrile.

      [Claim 2] A method according to claim 1, wherein the  
20        gas mixture excluding maleimide and acrylonitrile is composed of molecular oxygen and an inert gas.

      [Claim 3] A method according to claim 2, wherein said inert gas is nitrogen.

      [Claim 4] A method according to claim 1, wherein said  
25        acrylonitrile solution is heated to and retained in a range of 30 - 70°C.

      [Claim 5] A method according to claim 1, wherein the concentration of maleimide in the acrylonitrile solution is in the range of 40 - 80 wt. %.

30        [DETAILED DESCRIPTION OF THE INVENTION]

      [0001]

      [Field of Utilization in Industry] This invention

relates to a method for handling the acrylonitrile solution of a maleimide and more particularly to a method for safe and stable handling of a maleimide in the form of an acrylonitrile solution in the presence of a polymerization  
5 inhibitor.

[0002] The term "handling" as used in this invention means the transportation of the acrylonitrile solution of a maleimide as with tank lorries, the storage of the solution as in tanks, and the conveyance of the solution as with a  
10 piping system using pipes, valves, and nozzles (such as, for example, a supply line of the acrylonitrile solution of maleimide from the storage tank to the polymerization plant).

[0003]

[Prior Art] The maleimides which are represented by  
15 -phenyl maleimide are widely used as an agent for improving acrylonitrile-butadiene-styrene (ABS) resin, acrylonitrile-acrylic rubber-styrene (AAS) resin, acrylonitrile-styrene (AS) resin, and acrylonitrile-chlorinated polyethylene-styrene (ACS) resin  
20 in resistance to heat in the copolymerization of monomers forming such resins. They are also useful compounds as the raw materials for medicines and agricultural chemicals.

[0004] The maleimides which are solid at room temperature have been heretofore handled generally in such forms as powder,  
25 flakes, and tablets. The maleimides in such solid forms, however, yield to gradual pulverization in the course of transportation, emit impalpable powder in large quantities, and pose various problems such as deterioration of working atmospheres. The present applicant for patent, with the  
30 object of solving these problems, formerly proposed a method for safe and stable handling of a maleimide in the form of an acrylonitrile solution in the presence of a polymerization

inhibitor (JP-B-03-12057). Since this method of handling a maleimide in the form of an acrylonitrile solution is excellent in the point of having solved such problems as mentioned above, it is now being used extensively on the industrial scale.

5       [0005] As other ways of handling a maleimide in the form of an acrylonitrile solution, the present applicant for patent proposed a method of adjusting the acid content in the acrylonitrile solution of the maleimide to 0.3 wt. % (JP-A-64-61456) and a method of adjusting the acid content  
10   in the acrylonitrile solution of the maleimide to not more than 0.3 wt. % and requiring the presence of a polymerization inhibitor in the solution (JP-A-01-250348). These methods are directed toward preventing the acrylonitrile solution of the maleimide from corroding metallic materials and  
15   allowing safe and stable handling of the acrylonitrile solution as well.

      [0006] The present applicant for patent further proposed novel a method of handling a maleimide in a molten state (JP-A-03-209363 and JP-A-04-26673). This method is meant  
20   to handle a maleimide not in the form of an acrylonitrile solution but in a molten state at a temperature of not lower than the melting point of the maleimide and under the condition that the molecular oxygen content of the gas phase part be not more than 0.1 vol. % or not more than 10 vol. %.

25       [0007] It has been found that when a maleimide is handled in the form of an acrylonitrile solution, particularly in a large quantity on a commercial scale, by the method described in the official gazette of JP-A-03-12057, this handling possibly impairs the stability of the acrylonitrile solution  
30   by staining it or imparting turbidity to thereto. The acrylonitrile solution of the maleimide is possibly stained or rendered turbid, for example, when the acrylonitrile

solution of the maleimide is stored, for example, in a storage tank which was washed and has been left standing thence for a long time in the atmosphere of air or when a device used for some other purpose is diverted to a storage tank for the  
5 acrylonitrile solution. This phenomenon not merely impairs the market value of the acrylonitrile solution of the maleimide but brings about such situations as lowering the conversion of polymerization in the production of a resin aimed at, degrading such properties of the resin as resistance to heat,  
10 and blocking the feed line of the acrylonitrile solution from the storage tank to the polymerization system and rendering the production itself of the resin difficult.

[0008] The industry, therefore, has yearned for a method which solves such problems and permits safe and stable handling  
15 of a maleimide in the form of an acrylonitrile solution.

[0009] The present inventors, after pursuing a study in search of the cause of such adverse phenomena as staining and rendering turbid the acrylonitrile solution of a maleimide, have come to surmise that if the acrylonitrile solution happens  
20 to contain even a trace of oxide extraneously, the oxide will possibly decompose and form a polymer and eventually give rise to an opaque suspension in the acrylonitrile solution. Specifically, their inference is that if a storage tank left standing for a long time after a wash in an atmosphere of  
25 air is used or if a device used for some other purpose is diverted as a storage tank for the acrylonitrile solution of a maleimide, for example, the organic substance adhering to or persisting in the tank owing to insufficient cleaning will be oxidized with the oxygen in the air to form an organic  
30 oxide and consequently give rise to a polymer.

[0010] Further, this organic oxide is thought to promote this formation of the polymer to a further extent when the

acrylonitrile solution of the maleimide happens to have a monomer other than maleimide and acrylonitrile mingle therewith. Especially, since the acrylonitrile solution of a maleimide is used in the production of ABS resin, AS resin, etc. as mentioned above, the acrylonitrile solution possibly has styrene mingle therewith. This ingress of styrene accelerates the formation of the polymer and results in inciting an unforeseen situation in the actual handling of the acrylonitrile solution. Specifically, in the production of a styrene type resin, for example, this situation manifests as the counter flow of styrene from the styrene monomer feed line in the polymerization system to the storage tank of the acrylonitrile solution of the maleimide and the ingress of styrene into the storage tank from an inert gas seal line connected to the styrene tank and promotes the impartation of color or turbidness to the acrylonitrile solution of the maleimide or the formation of the polymer. It has been confirmed that this phenomenon is not limited to styrene but is shared by such aromatic vinyl compounds as  $\alpha$ -methyl styrene, p-methoxy styrene, p-methyl styrene, o, m, p-chlorostyrene, and p-bromostyrene.

[0011]

[Task to be fulfilled by the invention] This invention is directed toward solving such problems as mentioned above and providing a method for safe and stable handling of the acrylonitrile solution of a maleimide.

[0012] This invention is further directed toward providing a method for permitting the safe and stable handling of the acrylonitrile solution of the maleimide even when the acrylonitrile solution suffers ingress of a trace quantity of such an extraneous substance as a peroxide or an aromatic vinyl compound.

[0013]

[Means to fulfill the task] The present inventors, after making a diligent study in search of a method for effecting safe and stable handling of the acrylonitrile solution of a maleimide, have found that the molecular oxygen in the gas phase part of the acrylonitrile solution largely affects the stability of the acrylonitrile solution, the acrylonitrile solution is stable when the oxygen content in the gas phase part is adjusted in a specific range, and this stability is not impaired even when the solution happens to suffer ingress of a trace quantity of such an extraneous substance as a peroxide or an aromatic vinyl compound but is retained throughout the course of the handling. They have perfected this invention based on this knowledge. This knowledge has been totally unforeseeable in the light of the fact that acrylonitrile is generally stored in the atmosphere of nitrogen as prescribed in the disaster prevention guide.

[0014] To be specific, this invention concerns a method for handling a maleimide in the form of an acrylonitrile solution in the presence of a polymerization initiator, which method is characterized by adjusting the molecular oxygen content in the gas phase part of the acrylonitrile solution in the range of 0.01 - 10 vol. % based on the volume of the gas mixture excluding the maleimide and acrylonitrile. Now, this invention will be described in detail below.

[0015] As concrete examples of the maleimide which can be handled as contemplated by this invention, N-methyl maleimide, N-ethyl maleimide, N-hexyl maleimide, N-octyl maleimide, N-dodecyl maleimide, N-benzyl maleimide, N-cyclohexyl maleimide, N-phenyl maleimide, N-nitrophenyl maleimide, N-methoxyphenyl maleimide, N-methylphenyl maleimide, N-carboxyphenyl maleimide, N-hydroxylphenyl

maleimide, N-chlorophenyl maleimide, N-dimethylphenyl maleimide, N-cihlrophenyl maleimide, N-bromophenyl maleimide, N-cibromophenyl maleimide, N-trichlorophenyl maleimide, and N-tribromophenyl maleimide may be cited. The  
5 solution of a maleimide in acrylonitrile can be attained by an arbitrary method such as, for example, throwing the maleimide into acrylonitrile. Incidentally, this solution of the maleimide is preferred to be carried out at a temperature of not higher than the boiling point of acrylonitrile (78.5°C).  
10 Generally, the solution is preferred to be performed at a temperature in the range of 30 - 70°C because acrylonitrile has a high vapor pressure.

[0016] The concentration of the maleimide in the acrylonitrile solution does not need to be particularly  
15 restricted but may be properly decided in consideration of the temperature of handling, the manner of handling (transportation or storage, for example), the kind of a copolymer to be produced with the acrylonitrile solution, the method of production, the conditions of production, and  
20 the like. When the acrylonitrile solution is used in the production of a copolymer having a high maleimide content, for example, the concentration of the maleimide is possibly preferred to be high.

[0017] Generally in this invention, the concentration  
25 of the maleimide is in the range of 40 - 80 wt. % and the acrylonitrile solution containing the maleimide at a concentration in this range is handled at a temperature in the range of 30 - 70°C. According to this invention, even the acrylonitrile solution containing the maleimide at such  
30 a high concentration in the range of 60 - 80 wt. % can be handled safely and steadily as well. The polymerization inhibitor to be used in the acrylonitrile solution of the



maleimide may be any of the polymerization inhibitors heretofore known to the art. As concrete examples of the polymerization inhibitor, methoxybenzoquinone, p-methoxyphenol, phenothiazine, hydroquinone, alkylated  
5 diphenylamines, methylene blue, tert-butyl catechol, tert-butyl hydroquinone, zinc dimethyl dithiocarbamate, copper dimethyl dithiocarbamate, copper dibutyl dithiocarbamate, copper salicylate, thiodiisopropionic esters, mercaptobenzimidazole, triphenyl phosphite, alkyl  
10 phenols, and alkyl bisphenols may be cited. The kind of polymerization inhibitor may be properly selected in consideration of the kind of a copolymer to be produced, the method for producing this copolymer, and the kind of a polymerization initiator to be used.

15 [0018] Among other polymerization inhibitors cited above, p-methoxyphenol, tert-butyl catechol, and such hindered phenols as 2,4-bis(n-octylthio)6-(4-hydroxy-3,4-di-t-butyl anilino)-1,3,5-triazine, 2,2'-thiobis-(4-methyl-6-t-butyl phenyl), and triethylene  
20 glycol-bis[3-(3-t-butyl-5-methyl-4-hydroxyphenyl)propionate], especially p-methoxyphenol, are used particularly advantageously.

[0019] The quantity of the polymerization inhibitor to be used is in the range of 0.0001 - 1.0 wt. %, preferably  
25 0.001 - 0.1 wt. %, based on the weight of the maleimide.

[0020] According to this invention, when the acrylonitrile solution of a maleimide is to be handled, the oxygen content of the gas phase part of the acrylonitrile solution is fixed in the range of 0.01 - 10 vol. %, preferably  
30 0.1 - 8 vol. %, and more preferably 1 - 7 vol. %, based on the volume of the gas mixture excluding the maleimide and the acrylonitrile.

[0021] The gas mixture excluding the maleimide and the acrylonitrile mentioned above generally comprises molecular oxygen and inert gases such as nitrogen, carbon dioxide, helium and argon. Among other inert gases cited above, nitrogen is  
5 used particularly advantageously. As a preferred example of the gas mixture excluding the maleimide and the acrylonitrile, the mixed gas which is composed of molecular oxygen and nitrogen can be cited.

[0022] In short, in the preferred mode of this invention,  
10 the gas phase part of the acrylonitrile solution of a maleimide is formed of a gas mixture composed of maleimide, acrylonitrile, molecular oxygen, and nitrogen gas. The oxygen content in the mixed gas of molecular oxygen and nitrogen gas is in the range of 0.01 - 10 vol. %, preferably 0.1 - 8 vol. %, and  
15 more preferably 1 - 7 vol. %.

[0023] If the molecular oxygen content of the gas phase part (based on the volume of the gas mixture excluding the maleimide and the acrylonitrile; the same applies hereinafter) is smaller than 0.01 vol. %, the impartation  
20 of turbidness to the acrylonitrile solution of the maleimide or the formation of a polymer which is induced by the ingress of a monomer other than maleimide and acrylonitrile and the pollution of vessels including a storage tank will not be prevented. Conversely, if the molecular oxygen content of  
25 the gas phase part exceeds 10 vol. %, the excess will be at a disadvantage in tainting the acrylonitrile solution of the maleimide to the extent of increasing the Gardner color from 7 to 13 as shown in Control 3, for example. The color thus imparted to the acrylonitrile solution of the maleimide exerts  
30 serious adverse effects on the produced resin as by inducing the impartation of a color to the resin produced with the acrylonitrile solution or the degradation of the physical

properties of the resin. The commercial demerit brought about by the impartation of the color to the acrylonitrile solution is remarkable. A method which is capable of safely and steadily handling the acrylonitrile solution of a maleimide  
5 without inducing this impartation of a color, therefore, has been yearned for.

[0024] The method for fixing the molecular oxygen content of the gas phase part of the acrylonitrile solution of the maleimide in the range specified by this invention does not  
10 need to be particularly restricted but may be attained by displacing the gas phase part by using a mixed gas adjusted in advance to a prescribed molecular oxygen content with an inert gas such as nitrogen. The introduction of this mixed gas into the acrylonitrile solution of the maleimide by such  
15 an operation as stirring or bubbling is favorable because it enables the effects of this invention to be manifested fast.

[0025]

[Effect of the invention] The main effects of this  
20 invention are as cited below.

[0026] (1) It can handle the acrylonitrile solution of a maleimide safely and steadily.

[0027] (2) It can effectively prevent the impartation of a color or turbidness to the acrylonitrile solution or  
25 the formation of a polymer even under such a condition as forms an organic peroxide which is thought to be the cause for the impartation of a color or turbidness to the acrylonitrile solution of a maleimide or the formation of a polymer, namely under such a condition as suffers an organic  
30 substance originating in the insufficiency of cleaning to adhere to or persist in a storage tank which was left standing for a long time after a wash in an atmosphere of air and is

now used for the storage of the acrylonitrile solution or in a device which was used formerly for some other purpose and is now diverted to a storage tank for the acrylonitrile solution of a maleimide.

5        [0028] (3) It can effectively prevent the impartation of a color or turbidness to the acrylonitrile solution or the formation of a polymer even when a monomer other than maleimide and acrylonitrile, namely such an aromatic vinyl compound as styrene which seriously impairs the stability  
10 of the acrylonitrile solution, happens to mingle with the acrylonitrile solution of the maleimide.

      [0029] (4) It can steadily handle the acrylonitrile solution of a maleimide at a concentration in the range of 40 - 80 wt. % at a temperature in the range of 30 - 70°C as  
15 well.

      [0030] (5) It is incapable of impairing the market value of the product of the acrylonitrile solution of a maleimide because the acrylonitrile solution, even after a prolonged storage, will not yield to the impartation of a color or  
20 turbidness or the formation of a polymer.

      [0031] (6) It can produce a resin excelling in hue and physical properties by using the acrylonitrile solution because the acrylonitrile solution, even after a prolonged storage, will not yield to the impartation of a color or  
25 turbidness or the formation of a polymer.

[0032]

[Working examples] Now, this invention will be described more specifically below with reference to working examples. The term "oxygen content of the gas phase part" as used in  
30 the following working examples, controls, and tables is to be construed as based on the volume of the mixed gas excluding the maleimide and the acrylonitrile.

[0033] Example 1

In a 500-mL flask fitted with a stirrer and a condenser, 90 g of acrylonitrile and 10 mg of p-methoxy phenol as a polymerization inhibitor were placed. This flask was placed  
5 on a water bath and adjusted to an internal temperature (the temperature of the acrylonitrile solution) of 65°C by adjusting the temperature of the water bath. Then, when this solution was stirred and 210 g of N-phenyl maleimide crystals having an assay of 99.5% were added to the stirred solution, the  
10 N-phenyl maleimide crystals quickly dissolved and produced a perfectly clear yellow solution. When this solution was measured with a colorimeter (made by Tokyo Kodan K.K. and sold under the trademark designation of "Delta Colorimeter") for hue, it showed Gardner No. 7.

15 [0034] A test piece of stainless steel (SUS 304) measuring 20 mm × 40 mm × 2 mm was placed in the solution and then nitrogen gas containing 7 vol. % of molecular oxygen was supplied into the solution till the air in the gas phase part was displaced with the molecular oxygen-containing nitrogen gas and the  
20 flask was tightly sealed. The molecular oxygen content in the gas phase part was 7 vol. %.

[0035] When the flask containing the solution was subsequently retained at an inner temperature of 65°C for 60 days, the solution kept the clarify intact even after the  
25 60 days' standing. The hue of the solution was Gardner No. 8.

[0036] When this solution was evaporated to expel acrylonitrile, it produced vivid yellow crystals. When the crystals were analyzed by high-speed liquid chromatography  
30 for N-phenyl maleimide, the assay of the compound was found to be 99.5%. It showed absolutely no discernible sign of polymerization.

[0037] The results clearly indicate that the acrylonitrile solution of N-phenyl maleimide could be handled steadily in the vessel made of stainless steel.

[0038] The conditions for handling and the results are  
5 summarized respectively in Table 1 and Table 2.

[0039] Example 2

In the same flask as used in Example 1, 100 g of acrylonitrile, 3 g of styrene, and 10 mg of p-methoxy phenol as a polymerization inhibitor were placed. This flask was  
10 placed on a water bath and the inner temperature of the flask was set at 65°C by adjusting the temperature of the water bath. Then, when the solution was stirred and 200 g of -phenyl maleimide crystals having an assay of 99.5% were added to the stirred solution, the N-phenyl maleimide crystals were  
15 dissolved fast and produced a perfectly clear yellow solution. When this solution was tested for hue in the same manner as in Example 1, Gardner No. 7 was obtained.

[0040] The same test piece of stainless steel as used in Example 1 was placed in the solution, nitrogen gas containing  
20 7 vol. % of molecular oxygen was subsequently supplied to the gas phase part till the air in the gas phase part was displaced with the oxygen-containing nitrogen gas, and the flask was tightly sealed. The molecular oxygen content in the gas phase part was 7 vol. %.

25 [0041] When the solution was retained at an inner temperature of 65°C for 30 days, the solution kept the clarity intact and showed a hue of Gardner No. 8 even after the 30 days' standing.

[0042] When this solution was evaporated to expel  
30 acrylonitrile, it produced vivid yellow crystals. When the crystals were analyzed for the N-phenyl maleimide content in the same manner as in Example 1, the assay of the compound

was found to be 99.5%. It showed absolutely no discernible sign of polymerization.

[0043] The results clearly indicate that, even in the presence of styrene as an extraneous substance, the  
5 acrylonitrile solution of N-phenyl maleimide could be handled steadily in the vessel made of stainless steel.

[0044] The conditions for handling and the results are summarized respectively in Table 1 and Table 2.

[0045] Control 1

10 The displacement of the gas phase part was carried out by following the procedure of Example 2 while using nitrogen gas in the place of the molecular oxygen-containing nitrogen gas. When the gas phase part which had undergone the displacement was analyzed for gas composition by gas  
15 chromatography, it was found to have an oxygen content of 0.001 vol. %, a value indicating substantial absence of oxygen.

[0046] When the solution was subsequently retained at an inner temperature of 65°C, it turned into a turbid viscous  
20 solution on the 30<sup>th</sup> day of the standing. When this solution was added dropwise into 1 liter of methanol solution which was kept vigorously stirred, it precipitated a white polymer.

[0047] When the polymer was separated by filtering the solution, dried, and then weighed, the polymer content was  
25 found to be 3 wt. % based on the total weight of N-phenyl maleimide and acrylonitrile used at first. The results indicate that the solution clearly formed a polymer.

[0048] The conditions of handling and the results are respectively summarized in Table 3 and Table 4.

30 [0049] Examples 3 - 6

The handling of an acrylonitrile solution of a maleimide was carried out by following the procedure of Example 2 while

such conditions as the kind of maleimide, kind of polymerization inhibitor, and the molecular oxygen content of the gas phase part were changed as shown in Table 1. The conditions of handling and the results are respectively  
5 summarized in Table 1 and Table 2.

[0050] Controls 2 and 3

The handling of an acrylonitrile solution of a maleimide was carried out by following the procedure of Example 2 while  
10 such conditions as the kind of maleimide, kind of polymerization inhibitor, and the molecular oxygen content of the gas phase part were changed as shown in Table 3

[0051] The conditions of handling and the results are respectively summarized in Table 3 and Table 4.



[0052]

[Table 1]

Example	Maleimide		Stabilizer		Impurities		Oxygen content of gas phase part (*4)	Conditions of standing	
	Kind	Concentration (*1)	Kind	Quantity added (*2)	Kind	Concentration (*3)		Temperature (° C)	Duration (days)
1	N-phenyl maleimide	70/30	p-methoxy phenol	100	-	-	7	65	60
2	N-phenyl maleimide	67/33	p-methoxy phenol	100	Styrene	1	7	65	30
3	N-phenyl maleimide	50/50	p-methoxy phenol	100	α-methyl styrene	1	7	65	30
4	N-phenyl maleimide	50/50	TBC (*5)	100	Styrene	1	7	65	30
5	N-phenyl maleimide	70/30	p-methoxy phenol	100	Styrene	1	7	65	30
6	N-(O-methyl-phenyl) maleimide	50/50	TBC	100	Styrene	1	2	65	30

(\*1) Maleimide/acrylonitrile (wt. %)

(\*2) ppm based on maleimide

(\*3) Wt. % based on (maleimide + acrylonitrile)

(\*4) Vol. %

(\*5) p-tert-butyl catechol

[0053]

[Table 2]

Example	Appearance of solution		Gardner No.		Quantity of formed polymer (*7)	Assay of maleimide (*8)
	Before standing	After standing	Before standing	After standing		
1	Clear	Clear	7	8	-	99.5
2	Clear	Clear	7	8	-	99.5
3	Clear	Clear	7	8	-	99.5
4	Clear	Clear	7	8	-	99.5
5	Clear	Clear	7	8	-	99.5
6	Clear	Clear	7	8	-	99.5

(\*7) Wt. % based on (maleimide + acrylonitrile)

(\*8) Wt %.

[0054]

[Table 3]

Control	Maleimide		Stabilizer		Impurities		Oxygen content in gas phase part (*4)	Conditions of standing	
	Kind	Concentration (*1)	Kind	Quantity added (*2)	Kind	Concentration (*3)		Temperature (°C)	Duration (days)
1	N-phenyl maleimide	50/50	p-methoxy phenol	100	Styrene	1	0	65	3
2	N-phenyl maleimide	50/50	p-methoxy phenol	100	$\alpha$ -methyl styrene	1	0.001	65	4
3	N-phenyl maleimide	50/50	TBS (*5)	100	Styrene	1	15	65	30

(\*1), (\*2), (\*3), (\*4), (\*5): Same as in Table 1.

[0055]

[Table 4]

Control	Appearance of solution		Gardner No.		Quantity of formed polymer (*7)	Assay of maleimide (*8)
	Before standing	After standing	Before standing	After standing		
1	Clear	Turbid	7	-	3	-
2	Clear	Turbid	7	-	5	-
3	Clear	Clear	7	13	-	99.5

(\*7) , (\*8): Same as in Table 2

5 [0056] Example 7

A jacketed reaction vessel (measuring 200 L in volume and made of SUS 304) fitted with a stirrer and a condenser and used for the production of a maleic anhydride-styrene-methyl methacrylate polymer was washed  
10 with hot water and steam, dried in a stream of air at 100°C, and then diverted as a storage vessel for the acrylonitrile solution of N-phenyl maleimide.

[0057] The vessel was thoroughly displaced with nitrogen gas, then charged with 60 kg of acrylonitrile and 4.5 g of  
15 p-methoxy phenol, and heated to 50°C. Then, when 90 kg of N-phenyl maleimide was added to the solution and dissolved therein, it dissolved quickly and produced a thoroughly clear yellow solution. When the gas phase part was displaced with nitrogen gas and the outlet gas was analyzed for oxygen content,  
20 no oxygen was detected.

[0058] Then, when the acrylonitrile solution mentioned

above was retained in an atmosphere of nitrogen at 50°C, the acrylonitrile solution turned turbid after 5 hours' standing.

[0059] When the handling of the acrylonitrile solution was performed by following the procedure described above while  
5 using nitrogen gas containing 7 vol. % of molecular oxygen in the place of the nitrogen gas, the acrylonitrile solution kept the clarity intact even after 30 days' standing. This solution showed no discernible sign of turbidness.

[0060] It is noted from the results given above that  
10 the impartation of turbidness to the acrylonitrile solution which is thought to be induced by pollution of the vessel could be prevented perfectly by setting the molecular oxygen content of the gas phase part at 7 vol. %.

[0061]